GAU 1621

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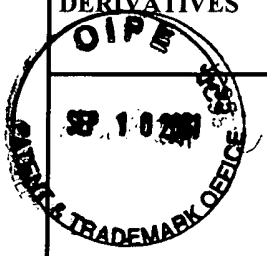
CERTIFICATE OF MAILING BY FIRST CLASS MAIL (37 CFR 1.8) Applicant(s): Kustov, et al.			Docket No. 8CL-7174A (GP1-0032-D)
Serial No. 09/682,010	Filing Date July 9, 2001	Examiner	Group Art Unit 1621

Invention: **PREPARATION OF CATALYSTS USEFUL IN THE PREPARATION OF PHENOL AND ITS DERIVATIVES**

#3



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Practitioner's Docket No. 8CL-7174A (GP1-0032-D) *PATENT*

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT:	Kustov, et al.	)	
		)	Group Art Unit:
SERIAL NUMBER:	09/682,010	)	1621
		)	
FILED:	July 9, 2001	)	
		)	Before the Examiner:
FOR:	PREPARATION OF CATALYSTS USEFUL IN THE PREPARATION OF PHENOL AND ITS DERIVATIVES)	)	

Commissioner for Patents
Washington, D.C. 20231

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SIR:

Applicant hereby submits the attached material references A-C which are considered to be proprietary, and Applicant requests that this material be considered under M.P.E.P. § 724.02. A Petition under 37 CFR § 1.59, and the fee therefore (37 CFR § 1.17(i)) to expunge the information if found not to be important to a reasonable Examiner in deciding whether to allow the Application to issue as a patent also accompanies this material.

Respectfully submitted,

KUSTOV, ET AL.

CANTOR COLBURN LLP
Applicants' Attorneys

By: Patricia DeSimone
Patricia DeSimone
Registration No. 48,137

Date: September 6, 2001
Customer No.: 23413
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I hereby certify that this correspondence was deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner for Patents, Washington, D.C. 20231 on

September 6, 2001
(Date of Deposit)

Tracy A. Sweatt
(Name of Person Mailing Paper)

Tracy A Sweatt 09/06/01
Signature Date

12 Nov 1997

To: File

From: John W. Fulmer

Subject : Monsanto (Solutia) - New information

Mr. Steve Sautman, a Lead Process Engineer of Monsanto, visited the GE Mt. Vernon site today (12 Nov) as a candidate for potential employment at GE. He is based in St. Louis. The topic of his seminar was "Optimization of the Benzene/N2O route to Phenol (BTOP)".

Mr. Sautman appears to have been a key member of the Monsanto (Solutia) development team during the 1995-97 period. His expertise is process design and process control optimization using "HYSIM"/Icarus modeling. During his seminar presentation Mr. Sautman was surprisingly free with technical information on the new "BTOP" route and even displayed transparencies of some rather detailed PFD's of their commercial process in Pensacola.

Here are my notes from the seminar. Some of this information conflicts with what we have learned earlier (either indirectly and directly) from Monsanto:

1. Mr. Sautman claims Monsanto has an issued U.S. patent on the new BTOP process. (I am not sure this is strictly true - I have seen only patents from their Russian partner, Boreskov Institute.)
2. Their N2O raw material is a waste gas from their adipic acid unit and a purification step is critical to remove NOx before it is used.
3. Commercial installation uses 4 fixed-bed parallel reactors, filled with a zeolite type catalyst. They operate three on-line while the other is in regeneration mode. Regenerate with air. Run time is 48hrs between regens, with regeneration time at 16 hours. Catalyst life is 18 months, after which they landfill.
4. Composition of reactor crude effluent is about 98 wt% phenol. Main by-products are (diols) hydroquinones. Benzene conversion per pass is 5-10%, with 70-80% selectivity on N2O and 90-95% selectivity on benzene.
5. Mr. Sautman claims they run the reaction with an <u>excess of benzene</u> and showed a PFD of the overall process with a large benzene recycle stream. (This statement seems to conflict with earlier information that Monsanto uses a N2O/benzene feed mole ratio of 4:1. I questioned Mr. Sautman about this but he remained firm that a large excess of benzene is employed.)

I did not ask many detailed questions and did not tell him about specific GE interest in this process. He may not know about the parallel ongoing GE-Zelinsky work on this route, although other people at Monsanto do.
J. Fulmer

11/12/97

PUMP 300 MM LB/YR

STEVE SAUTMAN – SOLUTIA

(LEAD PROCESS ENGINEER) (MONSANTO)

- HYDROXYLATION OF BZ/N_2O → PNL. (BTOP) ZEO. "Y"
- ? – US PATENT? CLAIMS MONSANTO HAS ONE.
- PURIFICATION OF N_2O NECESSARY.
  - REMOVE NOx FROM IT.
- FORM "DIOLS" BYPRODUCTS (
- 4 REACTORS / 3 ON-LINE (FIXED BED) 15' DIA
  - 1 – REGEN MODE

- RUN TIME – 48 HOURS BETWEEN REGENS – 10 HR REGEN TIME
- 18 MONTHS CAT LIFE
- LANDFILL
- 99% PURITY PNL CRUDE (HYDROQUINONES)
  - REACTOR EFFLUENT

USING "HYSIM" MODELING (ICARUS)

- N_2O/BZ
- TEMP
- RESIDENCE TIME

RUNNING WITH BZ EXCESS!

- REGEN: ADD O_2 TO BURN OFF.

- CONVERSION PER PASS: 5–10% BZ (SELECTIVITY 70–80% N_2O, 90–95% BZ)
  - RUNNING XS. BENZENE, HUGE BZ. RECYCLE.

PROPRIETARY MATERIAL

STEVE T. SAUTMAN

1054 S. Kingshighway Blvd. Apt. F, St. Louis, MO 63110
(314) 534-4519 (H)
314-694-4431 (W)

OBJECTIVE

A position where I can improve operations and manufacturing processes in order to better meet customer needs.

EDUCATION

JOHN M. OLIN SCHOOL OF BUSINESS, WASHINGTON UNIVERSITY - ST. LOUIS, MO

Master of Business Administration. December 1997. GPA 1.8/2.0. Evening Program.
Concentration in Operations & Manufacturing Management.
Independent Study in OMM to align manufacturing and marketing strategies for a product at work.

UNIVERSITY OF ILLINOIS AT URBANA-CHAMPAIGN

Bachelor of Science in Chemical Engineering - May 1993. GPA 4.6/5.0.
Graduated with Distinction in Curriculum. Member of Tau Beta Pi. Received Dean's List for 4 Semesters.

PROFESSIONAL EXPERIENCE

MONSANTO - Chemicals Company Engineering — St. Louis, MO

Lead Process Engineer for $110M New Technology Project — February 1997 - Present

- Conceptualize process design and evaluate operational and economical impact of alternatives.
- Direct internal and contracted people in the development and engineering phases of projects.
- Develop a 5 year Facility Plan for the Adipic Acid process to align its manufacturing and marketing strategies concerning capacity, reliability, quality, and technology as a member of the business team.
- Coach new process engineers on project development, economics, equipment modeling and design.

Lead Process Engineer for $10M Adipic Acid Project — June 1995 - Jan. 1997

- Developed project scope and premises by analyzing alternatives for a 10% capacity expansion project.
- Led and coordinated the process front-end engineering design by identifying issues and prioritizing tasks among engineering, manufacturing, technology, utilities, and contractors.
- Guided the technology development of a new reactor design and of using a new column packing type.
- Optimized the commercial plant design of the new direct hydroxylation of benzene to phenol process by linking its reaction conditions to its associated capital and operating costs.

Process Engineer — June 1993 - May 1995

- Identified bottlenecks for Nylon Intermediates expansion projects by creating simulation models.
- Designed process flow diagrams, piping and instrumentation diagrams, and equipment.
- Improved manufacturing processes through projects by eliminating and simplifying steps.
- Developed, tracked, and reported the Key Performance Indicators for Fiber Engineering projects.

DOW CHEMICAL COMPANY — Midland, MI

Cooperative Education Program — Sep. '89 - May '92 (5 Terms - 19 months actual work)

- Implemented cardboard recycling program in manufacturing unit.
- Applied Statistical Process Control charting to improve the particle size consistency of PellaDow.
- Managed the re-insulation of Anhydrous Ammonia tanks in chemical distribution unit.
- Optimized reaction conditions for a new herbicide using design of experiments.
- Tested and correlated latex properties with their carpet end-use characteristics.

SKILLS AND PROFESSIONAL AFFILIATIONS

- Technical: Hysys, Hycon, HTRI, Column Packing & Tray Design, Icarus Process Evaluator.
- Software: Windows 95, Word, Excel, Lotus Notes, PowerPoint, Visio.
- Interests: Biking, Racquetball, Traveling, Skiing, Dance St. Louis, Literature, Painting.
- Engineer-In-Training. Member of American Institute of Chemical Engineers.

F-O-R-T-U-N-E OF S.W. INDIANA
MT. VERNON, IN 47620
(812) 838-6636
CONSULTANT: Gary Fox
DATE: 11-3-97

Monsanto

THE CHEMICAL GROUP
P.O. Box 97
Gonzalez, FL 32560-0097
Phone: (904) 968-7000

April 14, 1997

John Fulmer
Manager - Lexan Quality Technology
Plastic Manufacturing Div.
Bldg. 40
1 LEXAN Lane
Mt. Vernon, IN 47620-9364

Dear John Fulmer:

Enclosed are two sample of Monsanto's phenol produced at the Pensacola Pilot Plant. The phenol is approximately 100 ppm level of impurities; the two primary impurities are 10 ppm O-Cresol and 60 ppm Naphthalene. Our target is to produce phenol under the 100 ppm of total impurities, but wished to get you these samples as quickly as possible. We have not set up our analytical lab for routine phenol analysis and would appreciate your analysis of the phenol samples to confirm our results and a comparison to GE's phenol. My number in Pensacola is (904)-968-7428.

Sincerely,

Chris Buechler

Christopher R. Buechler
Monsanto
P.O. Box 97
Gonzalez, FL 32560-0097

PROPRIETARY MATERIAL

REPORT: 7810 CHANNEL: 23 IMPURITIES IN PHENOL

SAMPLE: PHDI97-31RN7 INJECTED AT 9:24:04 ON APR 9, 1997

ISTD METHOD: M23PH1 SEQ: S23BZ1 SUBSQ/SAMP: 8/116 BTL: 1

SL-WDTH	MV/MIN	DELAY	MIN-AR	BUNCH
.125	.100	2.60	50	AUTO

SUP-UNK	DVT	ID-LVL	REF-RTW	%RTW	%DIL-F	ISO
NO	0.00	0	.050	-.120	10000.	NO

ACTUAL RUN TIME: 30.008 MINUTES

ISTD-RATIO: 4.326 CA STD-AMT: .0437 SAMP-AMT: 1.0091

RT	ITM	FACTOR	AREA		CA	NAME
5.02	5.02#	1.00000	1596212	BB		&INTERNAL STANDARD
9.33	9.33#	0.00000	25955152	BS	0.000	#PHO
9.66	9.66#	.98764	509	TT	.001	#O-CRESOL
10.26	10.26#	.76765	2818	TB	.006	#NAPHTHALENE
15.12		1.00000	772	BB	.002	
20.67		1.00000	156	BB	4.2E- 4	

TOTAL AREA = 27555615 TOTAL CA = .010

PROCESSED DATA FILE: S23116 RAW DATA FILE: H23116

31 January 1997

To: Greg Chambers
Jean Heuschen
Wayne Hewett
Wendell Miller

From: John Fulmer

Subject: Trip Report to Monsanto Company - 30 Jan 1997
New Phenol Technology

A meeting was held with Monsanto representatives at their Pensacola, FL plant site on 30 Jan 1997, to discuss their commercialization plans for their newly announced phenol plant. Those present were:

Charles Weidhas - Monsanto Product Director. Intermediates
A.M. Patterson - Monsanto Manager of Fiber Intermediates Technology
A. K. Uriarte - Monsanto Chemist and R&D Manager, Intermediates
Christopher Buechler - Monsanto Process Engineer
Wendell C. Miller - GE Plastics
John W. Fulmer - GE Plastics

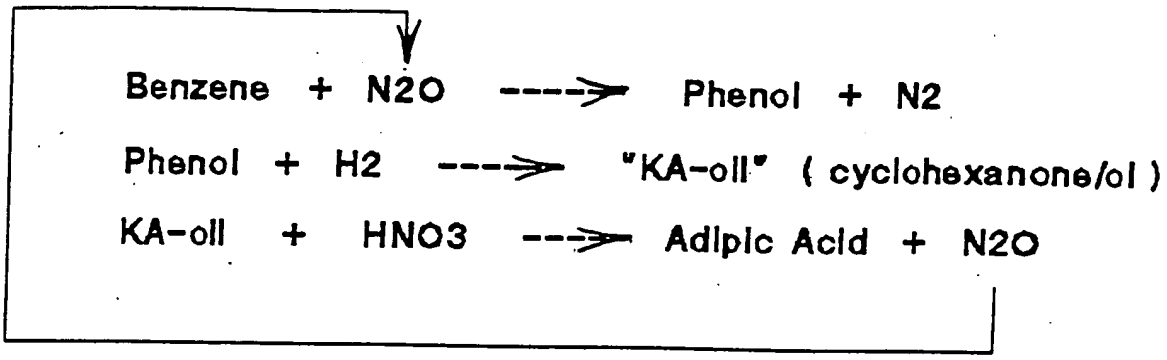
A technical secrecy agreement was not signed in advance of this meeting, because it was decided that any future GE role would most likely be as a purchaser of phenol rather than a licensee or equity partner in a new plant. Therefore confidential details of the new Monsanto benzene-to-phenol technology were not requested nor received as this time. A summary of my notes follows:

Current Status of Monsanto Project

Monsanto has been working with Boreskov Institute in Russia for past 3 years to develop a new phenol process based on N2O oxidation of benzene. Initial interest by Monsanto stemmed from the need to abate N2O emissions from their adipic acid units. Boreskov developed the new reaction technology and Monsanto developed the back-end phenol purification technology. Monsanto uses the acronym "BTOP" (benzene to phenol) for this technology. Monsanto has built a pilot unit at Pensacola and has been operating it since June 1997.

Monsanto schedule is to have a 300MM lb/yr commercial unit operating at Penscola in 4 Qtr 1999. Their schedule is:
2Qtr 1997 - Complete BEP
1Qtr 1998 - Complete Detailed Engineering
2Qtr 1998 - Break Ground
4Qtr 1999 - Start up

Monsanto would plan to use 100MM lb/yr phenol to meet their own internal needs, with 200MM lb/yr available for merchant sale. The Monsanto flow of chemicals in the adipic chain is shown below:



The new BTOP phenol technology will purify and recover waste N2O from the adipic acid units and react it with purchased benzene to form phenol, thus closing the above loop. Currently Monsanto purchases cyclohexane as raw material for the KA-oil using Halcon technology. The old KA-oil unit is out of capacity and has poor economics compared to phenol route.

Monsanto stated that one mole of N2O is generated per mole adipic acid, and that this ratio cannot be changed by varying conditions within the adipic unit. Four billion pounds of adipic acid is produced worldwide according to Monsanto. They claim enough by-product N2O is generated world-wide to support manufacture of one billion pound/yr of phenol via this route. Interestingly enough, Monsanto is also considering building an "on-purpose" N2O plant, using purchased ammonia as raw material. This indicates that purification of the N2O to the required quality from the stack gas containing NO and NO2 is not easy. Monsanto stated that the waste N2O is not really a "free" raw material.

Monsanto states that the investment for their new 300MM lb/yr plant will exceed $100MM, but that this capital is only half of what a plant based on cumene would cost. Monsanto touts this as the big advantage for licensing their technology. Manufacturing cost comparisons on the two processes (cumene vs. BTOP) are quite close however and no big economic advantage appears to exist in favor of BTOP, even when waste N2O is utilized.

Monsanto has not yet received Board appoval of the full capital funds required for the new phenol plant. Some uncertainty exists whether Monsanto will actually go ahead with the plant particularly in light of their major corporate reorganization currently underway with de-emphasis on chemicals.

Phenol Quality from BTOP Proc ss

A one-gallon sample of phenol produced from the pilot unit was shown to us. Color was water-white with a slight off-odor. Their GC analysis showed 230 ppm total impurities, mainly benzene, toluene, cresols and with 1-2 ppm hydroquinone. They had no information on freeze point, carbonyls, SAD or the other analytical tests commonly run on phenol. This sample had been recently produced (Jan 21) and was only one week old so color stability over time is unknown. The Pensacola people are not experts in phenol – they kept asking us for information regarding phenol handling and analytical . Monsanto would like to have a sample of GE Mt. Vernon phenol to benchmark against and also asked us if they could visit our phenol laboratory.

Monsanto was secretive about their pilot unit. They would not disclose its capacity or size other than to say it was a full-blown unit being 4 stories high. A newspaper photo of the pilot unit was given to us. They did not disclose if the pilot unit was using recovered N2O or purchased N2O. Monsanto stated that they had been running the purification portion of their pilot unit only three weeks and had very little information on final product quality. It is evident Monsanto is still optimizing their BTOP process and do not have a final design at this point.

Action Plan

Monsanto will send J. Fulmer two 16 oz samples of their BTOP phenol for GE analysis. If quality looks good, GE may then request larger samples for GE pilot synthesis of BPA to prove suitablity. This qualification process will take considerable GE time and effort and such a study must be carefully conducted to insure that good quality BPA will result and process issues such as potential IER catalyst poisoning are evaluated. It must be emphasized that the BTOP phenol process uses totally new chemistry and new impurities will exist in the phenol which possibly could act adversely in downstream users BPA/polycarbonate/PPO. Also, ISO9000 and Best Practices will require GE to notify plastics customers of a major process change of this nature, if we were to change to a new source of phenol.

W. Miller will issue separate meeting minutes focusing on the commercial aspects of the 30 January meeting.

J . W. Fulmer

CHEMICAL PROFILE

ADIPIC ACID

April 1, 1996

PRODUCER	CAPACITY*
AlliedSignal, Hopewell, Va.	40
DuPont, Orange, Tex.	400
DuPont, Victoria, Tex.	700
DuPont Canada, Maitland, Ontario	300
Monsanto, Pensacola, Fla.	600
Total	2,040

***Millions of pounds per year. Allied uses phenol as a feedstock and sells adipic acid on the merchant market. DuPont and Monsanto use cyclohexane feedstock and have captive requirements for nylon 6/6 manufacture. Monsanto is adding an undisclosed amount of acid through debottlenecking. Profile last published 11/6/95; this revision 4/1/96.**

DEMAND

1995: 1.85 billion pounds; **1996:** 1.9 billion pounds; **2000:** 2.1 billion pounds (Demand is for the US and includes exports, which were 155 million pounds in 1995, but not imports, which were 94 million pounds.)

GROWTH

Historical (1986-1995): 2 percent per year; **future:** 2 to 3 percent per year through 2000.

PRICE

Historical (1981-1996): High, 69.5c. per pound, list, resin grade bulk, hopper cars, frt. equald.; low, 50.5c. per pound, same basis. **Current:** 69.5c. per pound, same basis; 73.5c. per pound, packages, f.o.b., frt. equald.

USES

Nylon 6/6, 85 percent (fibers, 75 percent; resins, 10 percent); polyurethane resins, 8 percent; plasticizers, 3 percent; miscellaneous, including unsaturated polyester resins and food applications, 4 percent.

STRENGTH

Nylon fibers remain strong worldwide, with the export market growing in the Asia-Pacific area. In late 1995, DuPont brought on stream a 220-million-pound facility in Singapore to help cover demand in that area. Nylon resins are growing in automotive applications.

WEAKNESS

The market is very tight, but will be relieved when new worldwide capacity comes on line. Imports into the US are strong.

OUTLOOK

Worldwide demand growth for nylon will put continued stress on available adipic acid capacity until prices reach reinvestment levels. Growth is forecast for the next three years for nylon fibers and resins, and in polyurethanes and plasticizers. New uses are expected to emerge.

(212) 931-2762
ATT: JOHN FULMER, GENERAL ELECTRIC – NY REQUESTED
N. VAN, CMR 12/6/96